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Puck Passive Loop Seal

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ABSTRACT

Sandia National Laboratories is advancing technical capabilities used in passive loop seals. The "Puck" seal uses a set of International Atomic Energy Agency (IAEA) guidelines for new passive loop seals published in 2020 as a design guide. The seal is based on oxygen sensitive materials encased in an oxygen impermeable 3-D printed acrylate shell, is monolithic rather than 2-part, incorporates self-capturing wire features, contains colored water beads and bubbles formed during processing as unique identifiers, and visually indicates tamper (whether from seal body penetration or from seal wire removal) by irreversibly changing the seal body from multi-colored to black. Sandia has developed prototype seals that have undergone environmental testing and is currently modifying the design based on that testing. Further, Sandia is working together with Oak Ridge National Laboratory (ORNL) on another seal version, Puck/SAW, that uses the same seal body and incorporates a surface acoustic wave (SAW) chip that can provide a unique identifier upon standoff interrogation and monitor wire continuity. This paper will provide details on the design, development, and environmental testing of Puck prototypes.

INTRODUCTION

Tamper-indicating devices (TIDs, or seals) are deployed in many regimes including international nuclear safeguards, arms control, domestic security, and even commercial products to ensure that monitored items are not accessed without detection. A familiar commercial example of a tamper-indicating seal is the foil and/or plastic seals under the lids of medication bottles, which protect the consumer from product adulteration. Notably, the TID does not prevent access to the container's contents but is useful in alerting to potentially nefarious access. Due to the continual advancement of adversary capabilities, TIDs should also continually advance technically. New TID technologies may provide enhanced effectiveness (e.g., advanced tamper-indication or unique identifiers) and efficiency (e.g., verification in less time, more obvious tamper features, easier installation and maintenance); furthermore, the IAEA has specifically expressed its desire for advanced seals/TIDs.

Passive loop seals are extensively used in international safeguards to maintain the continuity of knowledge of declared material and equipment. While there is a plethora of loop seals available commercially, the IAEA has extremely stringent requirements that are not met by commercial offerings. One of those requirements is that the unique identity of the device can be proven to ensure that it is not a counterfeited replacement; the identity element of the seal must be non-reproduceable and cannot be replicated, but can be measured and verified. Another requirement is that the seal employs tamper-indicating features such that the seal wire cannot be removed and replaced (hence allowing access to the monitored item) without detection. The most ubiquitous loop seal deployed by the IAEA is the metal cup seal (also called CAPS) – a small, robust, simple

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device that captures sealing wire between two metal cup halves (Figure 1, left). Unique identification characteristics (i.e., ID number and randomized scratch patterns) are manually added to the metal cup seal prior to being deployed to the field. Significant resources (i.e., time, effort, cost) are required to verify the identity and integrity of the device post-mortem after seal removal and return to IAEA Headquarters (HQ). A best practice for seal programs is to periodically replace individual seals; the longer an installed seal is used in the field, the longer an adversary has to plan an attack and take advantage of a tampered container. Furthermore, the longer a seal design is utilized, the longer an adversary can take advantage of any discovered weaknesses of the seal technology that allows nefarious access to go unrecognized, thus defeating the purpose of the TID. As the simple metal cup seal has been utilized for over 45 years, an effort has been underway to replace various IAEA seals, particularly to create an economical seal that combines robust tamper-indicating mechanisms with unique identification (UID) characteristic materials.

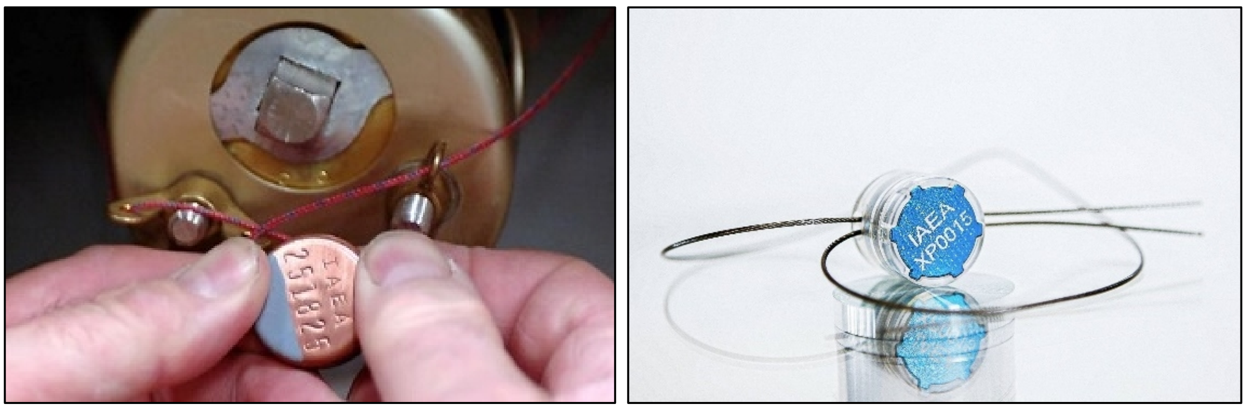

Figure 1: (Left) Metal cup seal [1]; (Right) Field Verifiable Passive Loop Seal, FVPS [2].

The IAEA has been seeking a replacement for the metal cup seal for many years and has identified a need for a passive loop seal with general requirements including in-situ verification, minimal use of external tools, unique identification, and tamper-indication of the seal body; the IAEA published a set of approximately 40 seal requirements in mid-2020.[3] We set out to use these published seal requirements and our significant subject matter expertise in tamper indication and seals to research, design, develop, and environmentally test a new passive loop seal. The impact is improved effectiveness (i.e., tamper-indication of seal body, inherent unique identifiers) and efficiency (i.e., quick tamper detection through visually obvious tamper responses, easy installation with minimal external tools required) providing benefit to the IAEA and to other regimes utilizing seals. We do note that the IAEA has developed a new passive loop seal very recently (first sharing details at the 2022 Safeguards Symposium) based on its requirements; this Field Verifiable Passive Loop Seal (FVPS, Figure 1, right) was designed and developed during the same time period that our project was proposed and funded. However, we have designed and are developing features that differ from the FVPS that provide value to advancing technical capabilities.

SEAL COMPONENTS AND DESIGN FEATURES

The most novel component of the Puck is the sensing mixture that occupies the interior volume of the Puck shell, which exhibits a visibly obvious indication of tamper when exposed to oxygen. The change of color is accomplished by the incorporation of a colorless chemical called L-3,4-

dihydroxyphenylalanine (L-DOPA). When dissolved in a basic aqueous solution, L-DOPA has a unique property of polymerizing into a brown melanin-like structure when it reacts with atmospheric levels of molecular oxygen. Thus, exposure to air induces a color change of the solution from pale yellow to dark brown. In order to exploit this behavior for uses in TIDs, we found that the oxygen-sensitive solution can be absorbed by commercial multi-colored water beads (made of sodium polyacrylate, the water-absorbing material used in diapers). This allows the oxygen-reactive chemical to be incorporated within solid water beads that are easy to process and can be easily contained within the body of Puck seals. Thus, when the water beads are loaded with the basic L-DOPA solution, they become active tamper indicators by undergoing a drastic, visibly obvious color change from multi-colored to black upon exposure to air (Figure 2).

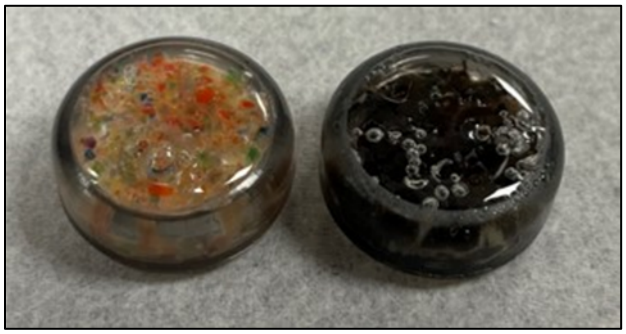

Figure 2: Two early Puck prototypes composed of 3D printed body, interior sensing mixture, and epoxy poured-in lid to provide an oxygen impermeable shell. (Left) Puck coupon without an intentional breach of air-tight outer shell (i.e., un-tampered); (Right) Puck coupon with an intentional breach of air-tight outer shell (i.e., after simulated tamper attack).

We have experimented with various versions of sensing mixtures containing water beads and the oxygen-sensitive L-DOPA solution. For example, we investigated the effect of bead size on both performance and visual impact; instead of using whole beads, we use a commercial blender to chop beads into smaller pieces, since they grow significantly upon absorption of the L-DOPA solution. Since the Pucks are somewhat small relative to a fully grown water bead, reducing the bead size before Puck assembly allows for many small bead pieces to be packed into each Puck body, which affords a very effective UID aspect, where each individual Puck inherently demonstrates a different orientation of differently colored bead pieces (Figure 2). Inclusion of silicone within the sensing mixture acts as a filler to dilute the amount of beads used to fill the Puck body. The visual effect is that the beads are separated from each other, giving more visible, separated colored pieces, which further enhances the UID aspect. Furthermore, silicone is relatively oxygen-permeable, so oxygen is able to diffuse through the silicone filler and react completely with all soaked bead pieces within the Puck body. After pouring the silicone-containing sensing mixture into the Puck body, it takes approximately 24 hours for the silicone to fully cure – after this point, the sensing mixture is somewhat solidified inside the Puck body, which prevents shifting and movement of the bead pieces within the Puck body. Some of the different sensing mixtures will be discussed in more detail in the “Aging” section below.

After the concept of L-DOPA as a tamper indicator was established (through previously funded work [4]), we chose to take a systematic approach in the development of prototypes for the envisioned Puck passive loop seal. Each component was individually incorporated and optimized before adding an additional component in order to isolate any undesirable effects from each new design feature. For each new feature, several replicate Pucks were assembled inside an air-free glovebox, then exposed to air (removed from the glovebox), and observed for color change. Most false positives (i.e., observations of rapid and extreme darkening from multi-colored to black with no intentional tamper simulation) were noticeable within the first 0-2 hours; each set of Pucks were observed for at least 3-7 days to record occurrences of false positives. For trial batches with high numbers of false positives, troubleshooting was performed to minimize false positive occurrence before proceeding with the addition of the next design feature. For example, when 3D printed lid parts were first added as replacement for poured-in epoxy-set lids, we discovered that we needed a locking mechanism to prevent the lid from rising up off the Puck body part during the curing process of the internal sensing mixture. Additionally, with the preliminary addition of the plug pieces, we determined that epoxy was needed to affix the plugs in place in order to maintain an air-tight seal.

The completed Puck prototype is assembled from several 3D-printed components consisting of an outer shell (body and lid pieces), plugs that are sealed into the shell at each location for wire punctures (2 entry and 2 exit points), and internal wire capture components (Figure 3). Each of these components will be discussed in detail in the remainder of this section. The interior volume of the Puck is filled with a sensing mixture that undergoes a visibly obvious and irreversible color change from multi-colored to black upon exposure to oxygen (Figure 2), which is intended to alert an inspector to a potential tamper attack. Thus, the outer shell of the Puck is designed to prevent oxygen from entering the Puck interior and interacting with the oxygen-reactive sensing mixture, except in the event of tamper.

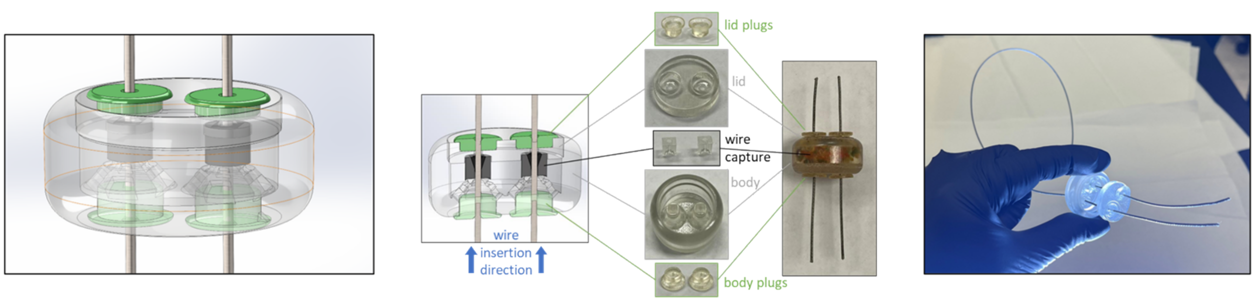

Figure 3: (Left) 3D transparent CAD image of Puck with all components shown; (Middle) CAD image showing cross section of Puck, individual 3D printed components, and fully assembled Puck with sensing mixture; (Right) unassembled Puck without sensing mixture showing wire loop inserted through Puck loop seal.

The outer shell of the Puck is composed from two parts – the lid piece and body piece (Figure 3) – that are both 3D printed from a PolyJet photopolymer material (UV curable resin), VeroClear, that is similar to polymethylmethacrylate (PMMA, acrylic). The parts are stiff, robust, and transparent, imparting ease of handling, impact resistance, and the ability to see through the Puck body so that interior contents (i.e., colored sensing mixture) are easily visible. The key functionality of the outer shell is to provide a relatively oxygen-impermeable shield around the sensing mixture that fills the interior of the Puck, in order to prevent oxygen-induced color change in the absence of a tamper attack. During assembly, the Puck lid piece is sealed into the Puck body piece by applying a small amount of epoxy to the outer rim of the Puck body, which glues the Puck lid in place, and allows the Puck device to maintain an air-tight seal as a single monolithic object.

Once assembled, the outer dimensions of the Puck body are approximately 30 mm (diameter) by 15 mm (height).

The Puck design prominently features a twin pair of parallel wire-entry columns, which each consist of a body plug, collapsing cone, wire capture, and lid plug (in order of the direction of wire insertion, blue arrow in Figure 3). The pair of collapsing cone features are both printed with the Puck body piece, and do not require any assembly. Each collapsing cone is a conical feature that has open “cut-out” channels along the sloped edge with given dimensions such that a pressure or force (i.e., tugging the wire with enough strength) will cause the cone to collapse and result in structural deformation, which allows air to enter the Puck body and react with the oxygen-sensitive sensing mixture, resulting in a visibly obvious color change that indicates tamper.

Sitting above the collapsing cone (Figure 3) is a wire capture feature that functions similarly to a zip tie capture by allowing the wire to only be inserted through the Puck body in a single direction. The wire capture feature is also 3D printed out of VeroClear material and is inserted into a cavity above the collapsing cone during Puck assembly. Small grooves or teeth effectively grab the wire such that the wire can easily slide through the device in one direction (i.e. to cinch the looping wire around an object or container lid), but the teeth prevent the wire from slipping or being easily pulled in the opposite direction (i.e. to pull the Puck off of the wire-looped monitored container). Thus, the wire capture feature functions to prevent nefarious removal of the Puck after installation, and circumvents the need for any external tool during installation since the feature is internal and self-contained.

Each Puck contains 4 plugs – 2 lid plugs and 2 body plugs, which have slightly different designs – to allow for wire penetration through the Puck. The plugs are 3D printed from a PolyJet photopolymer material, Agilus30, that is similar to rubber. The parts are soft and somewhat elastomeric, allowing the plug pieces to be punctured by a wire (even with a relatively blunt wire end), but also allows a sufficient seal around the wire after insertion to prevent air from entering the Puck body and reacting with the oxygen-sensitive sensing mixture. This duality in function represents the most difficult design challenge for the intended application of the Puck: to allow wires to be inserted through the Puck body (for the Puck to act as a passive loop seal), but also simultaneously to prevent air from entering the Puck body during expected installation so that the sensing mixture filling the interior of the Puck will remain active, to be able to change color if air is later introduced, and signal a tamper event. Notably, the plug design features a flange that sits on the outer surface of the Puck (i.e., on top of the Puck lid or on bottom of the Puck body; Figure 3) to ensure that the plugs are consistently inserted to the same depth for each Puck; the flange also provides additional surface area along which a small amount of epoxy is applied in order to permanently seal each plug to the Puck lid or body. Additionally, each plug has a guide hole, which is imparted by the 3D printed design, and allows easy insertion of the wire exactly in the middle of the plug. In order to maintain consistency with the currently deployed metal cup seals, we have used braided wire (18-8 SS, 7x19) coated with a plastic (FEP) sheath with outer diameter of 1.24 mm.

After ample iterations of designs and testing for false positives, we arrived at a version of the Puck prototype (Figure 4) that we felt was satisfactorily sufficient to move on to further, more specialized testing. This version was used for the testing procedures described in the following section. Importantly, out of 83 Pucks that were assembled for further testing, only 1 Puck was counted as a false positive after removal from the air-free glovebox environment. After wire insertion, 4 additional Pucks triggered an unintended color changing response and were not used in the test set. Furthermore, we demonstrated that wire removal from these Pucks did, as desired, trigger a visually obvious color change of the sensing mixture to black.

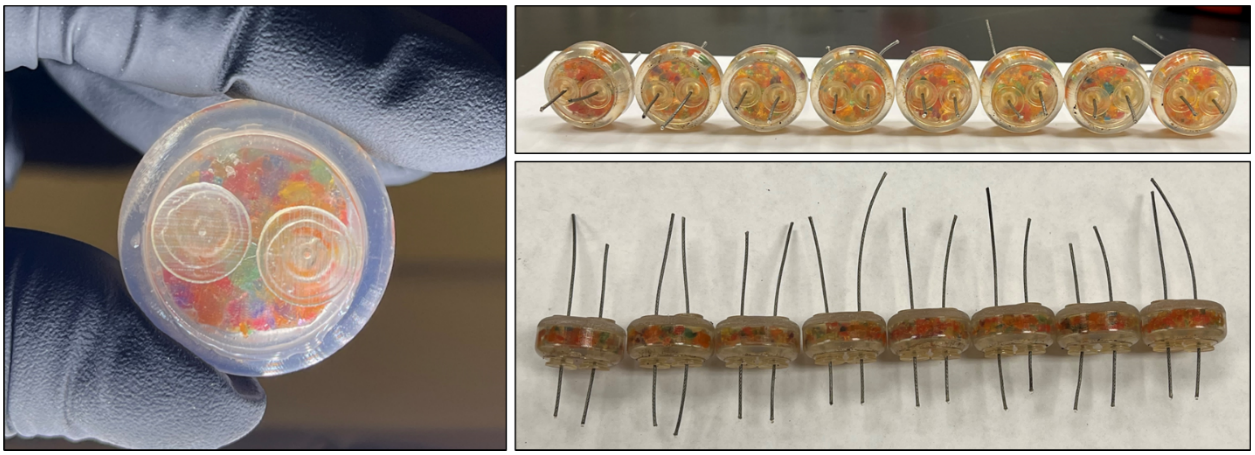

Figure 4: (Left) Assembled version of Puck prototype used for further testing. (Right) Multiple Pucks after wire insertions to show variety of bead distribution as UID feature.

TESTING

Our testing protocols were chosen based on the guidelines provided by the IAEA request for an updated passive loop seal.[3,5] The requests for mechanical stability include for the seal body to remain intact after being dropped from a height of 1.2 m, and to remain intact after being stepped on by a person; the Puck was found to exhibit no indications of damage after being dropped from 1, 2, and 3 m heights, and was similarly resilient to crush tests using forces up to 350 lbs both axially and cross-axis. The request for thermal stability was somewhat far-reaching, to withstand up to 3 years of exposure to a range from –50 to +150 °C. We exposed Pucks to either –30, 30, 40, 55, or 70 °C for up to 4 days, with removal of 2 Pucks from each temperature every 24 h; one of each pair of Pucks was drilled into (using a 1/16” drillbit) in order to confirm that the sensing mixture was still responsive to oxygen after thermal exposure (Figure 5). While Pucks were visibly unchanged after short-term exposure to –30, 30, and 40 °C, Pucks exposed to 55 and 70 °C demonstrated a visibly noticeable darkening which was not as extreme as a “false positive” event, but nonetheless undesirable. We referred to this observation as an aging effect, that we later determined takes place over longer time frames (i.e., 2-4 months) at room temperature in addition to short time periods (i.e., 1-4 days) at elevated temperatures. However, although this was initially disappointing, we think it is promising that the aged Pucks maintain the color-changing response mechanism; although they do darken over time, they still turn black within 1-2 hours of drilling into the outer Puck shell (Figure 5). We also performed UV exposure testing for up to 500 hours of alternating on/off cycles, with a similar procedure to thermal experiments, and observed similar aging affects.

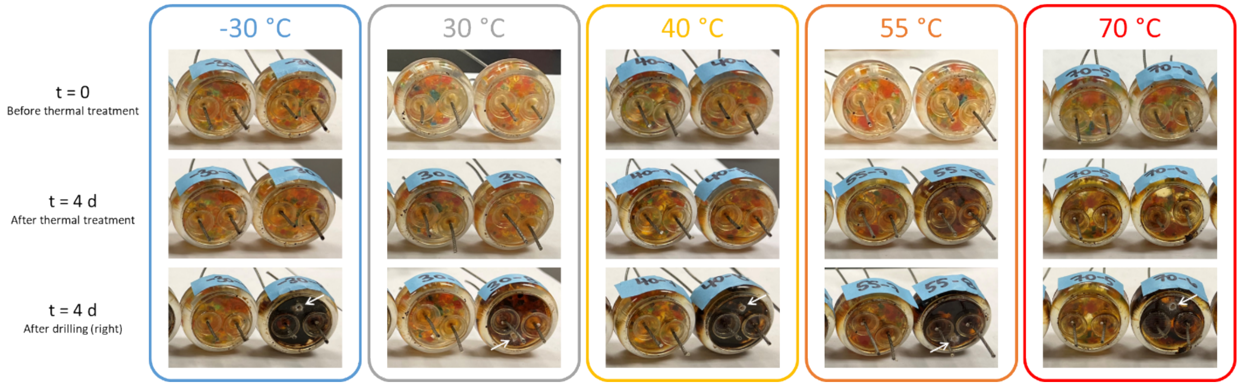

Figure 5: Selected Pucks before thermal treatment (top row, t = 0), after thermal treatment (middle row, t = 4 d), and after drilling the right-hand Puck in each sample pair (bottom row, white arrows indicate 1/16" drill holes).

In regard to the original envisioned application of Pucks as a potential replacement for the passive loop seals employed in nuclear facilities, we also performed radiation exposure testing on a series of Pucks (without wires inserted). Eight Pucks distributed among 3 different positions were stored inside a neutron howitzer containing 6 Ci of PuBe for 12 days and then evaluated visually. The set of Pucks displayed a range of visual changes similar to the previously observed aging effect that seemed to correlate with location such that Pucks positioned closer to the PuBe source demonstrated stronger aging effects. While Pucks directly touching the PuBe source were essentially completely blackened after 12 d, Pucks that were stored in side chambers (approximately 6-8 inches away from source, experiencing approximately 100 mrem/h) were visibly similar to a reference Puck that was not exposed to radiation. After being removed from the neutron howitzer, half of the Pucks were drilled to simulate a tamper event, and the Pucks stored in the side chambers were shown to have maintained their color changing response after radiation exposure, turning completely black within 30 min of drilling. Pucks stored directly under the PuBe source were so black from radiation exposure that they did not turn visibly blacker after being drilled.

AGING

As previously mentioned, after developing and optimizing a Puck prototype to study via extensive environmental testing, we discovered an undesirable aging phenomenon characterized by a darkening of the interior sensing mixture that occurs slowly over time at room temperature, and more quickly with exposure to elevated temperature, UV, or radiation. In order to investigate the cause of the aging effect (and potential mitigations to prevent or slow the aging process), we prepared 85 Pucks, with 5 replicate Pucks for each of 17 different design variations. Variations in designs included the use of Pucks with 0, 1, or 2 pairs of plugs to look for a correlation between number of plugs and extent of aging; additionally, Pucks were prepared with either no silicone incorporated within the interior sensing mixture, or using one of two different types of silicone in the sensing mixture to study the effect of sensing mixture composition; some designs included a post-assembly coating step involving the application of a clear nail polish over the outer plug surfaces to try to minimize air exposure to the outer plug surface; other designs involved layering the silicone and loaded beads within the Puck body to try to prevent interactions between the interior plug surface and the beads soaked in basic L-DOPA solution. From this study, we found that all Pucks containing any plugs underwent significant aging; the degree of aging varied among individual Pucks, but is extensive enough to result in Pucks that can become dark enough after 3-4 months (at ambient temperature and light exposure) to be visually dark enough to be mistaken as indicative of a tamper event. The only Pucks that remained visually "fresh" more than 4 months after assembly were those with no plugs (i.e., solid 3D printed lids). Unfortunately, a lack of plug would mean that these Pucks could not be deployed as passive loop seals (although the oxygen-sensitive concept could easily be applied for other applications). Furthermore, none of our design variations that included plugs showed significant improvement in terms of lessening or delaying the aging effect compared to the original prototype design.

A positive outcome from this study was that we were able to identify the plugs as the source for the aging effect. Our hypothesis is that the 3D printed plug material undergoes a change in composition over time – when freshly printed, the plug is squishy and malleable, however, several months later, the plugs are physically rigid and brittle. We believe that the plug is potentially becoming dried out over time, with some component of the plug material evaporating over time to cause its change in materials properties. This eventually results in the plugs becoming porous, which allows very small amounts of air to diffuse into the interior of the Puck and interact with the sensing mixture, such that the sensing mixture eventually reacts with enough oxygen so that the Puck visibly looks black after enough time has passed. We have ongoing studies underway to evaluate potential alternative rubber-like materials to use instead of the previously used Agilus30 plug material. Alternatively, we could assess a variety of design alterations using the Agilus30 plug material; for example, decreasing the height of the guide hole within the plug, or increasing the depth/thickness of the plug piece could potentially delay the aging effect, although we do not expect this to prevent aging altogether.

SUMMARY AND NEXT STEPS

We have designed, developed, and tested a prototype called Puck that includes a variety of features envisioned for the use as a passive loop seal, i.e., as a replacement for the metal cup seal currently deployed for monitoring of facilities for sensitive materials containment. The most prominent and novel design feature of Puck for use as a tamper indicating device is the interior sensing mixture which undergoes a color change from multi-colored to black upon exposure to oxygen, i.e., via adversarial penetration or compromise to the outer Puck shell, including removal of the looping wire. Elastomeric plugs inserted within the Puck shell body and lid allow puncturing of the Puck body by the looping wire while maintaining the air-tight seal. However, the plugs have been found to permit oxygen diffusion over long time periods (2-4 months at room temperature) such that the interior sensing mixture undesirably darkens over time. This aging phenomenon is also observed (to an accelerated extent) by thermal, UV, or radiation exposure. Next steps include evaluating alternative plug materials to improve long-term stability of the Puck color-changing tamper-indicating mechanism. Additionally, not discussed in this paper, Sandia is working with ORNL to develop a different version of the Puck loop seal that incorporates an aspect of active monitoring by encompassing a SAW chip and antenna within the sensing mixture and Puck shell.

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